

LEVEL



IMPROVEMENT OF PHOSPHORIC ACID

FUEL CELL STACKS

Interim Technical Report

May 1979

Prepared by

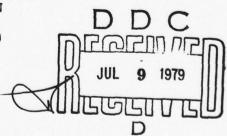
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Prepared for

U. S. Army Mobility Equipment Research and Development Command Fort Belvoir, VA 22060

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elf

stack was built and delivered to MERADCOM.

binder were used. Stacks were tested on hydrogen and simulated reformed methanol for up to 6,800 hours. An 80-cell (2.1kW)

SUMMARY

Phosphoric acid fuel cell component designs and stack assembly methods were evaluated. All work was with 5 in x 15 in cell dimensions, a size employed for constructing 2kW stacks for the 1.5kW methanol fuel cell powerplant.

Component development covered electrodes, the matrix, and the bipolar plate. The electrodes employed platinum on carbon catalyst at loadings of 0.3 to 0.9 grams/ft². A coating technique for preparing SiC matrices was developed. Bipolar plate mechanical and electrical characteristics were determined for a range of graphite-resin ratios.

The components were evaluated in multicell stacks. A total of 335 cells were assembled into 3, 10, and 80-cell stacks. Stack assembly techniques using both prefilled and dry matrices with wick filling were employed with equally good results. Both phenolic fiber (Kynol) and SiC matrices were used.

Stack testing was conducted with hydrogen and with simulated reformed methanol as fuel. Endurance testing of stacks was carried out for over 6,800 hrs, with optimum cell performance remaining above 640 mV at 100 ASF. An 80-cell, 2.1kW stack was assembled, tested with hydrogen fuel, and delivered to MERADCOM.

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1.0 INTRODUCTION

The purpose of this project is to improve performance and reliability of phosphoric acid fuel cell stacks. The effort is focused on improving bipolar plates, electrodes, matrices, and gas seals.

Stack component manufacturing methods were the subject of earlier work by Energy Research Corporation conducted under contract to USA MERADCOM. As a result of this effort, component manufacturing processes which are amenable to mass production methods were developed and evaluated in stack tests. (1)

On the present project, 51 stacks were built and tested employing improved bipolar plate designs and low loading platinum-on-carbon electrode catalyst. Silicon carbide as well as Kynol fiber was evaluated as a matrix material, and simplified stack assembly methods with reliable gas seals were tested.

Extensive endurance testing was undertaken with several 3-and 10-cell stacks. Six stacks were tested with hydrogen fuel beyond 4,000 hours, and an additional 10 stacks were tested beyond 2,000 hours. Several thousand hours of testing with simulated reformer product gas was also performed.

At the conclusion of this phase of the project, a 2.1 kW stack was constructed, tested, and delivered to MERADCOM.

2.0 COMPONENT DEVELOPMENT

2.1 Bipolar Plate Development

A molded bipolar gas distribution plate is the key component in a low cost air-cooled stack. Its design has a major bearing on performance characteristics and manufacturing economics of the phosphoric acid fuel cell.

In an earlier effort(1), techniques for molding bipolar plates using phenol-formaldehyde (PF) resins as binders for graphite were developed. The task on the current project was to examine various properties of the plates, to evaluate new plate configurations, and to obtain further life and performance data both under simulated and actual stack operating conditions.

⁽¹⁾ Contract DAAK02-74-C-0367, Final Report (1977)

2.1.1 Mechanical Properties

Mechanical testing of a bipolar plate sample was performed at Bridgeport Testing Laboratory, Inc. The sample was molded in the 5" x 15" bipolar plate mold using flat mold faces and had the composition 32% Colloid 8440 resin, 50% Asbury A99 graphite, and 18% Asbury 850 graphite. Molding conditions were 4300 psi at 340°F. The thickness of the plate sample was .165 in. Results of the mechanical tests are presented below in Table I.

TABLE I

MECHANICAL PROPERTIES OF BIPOLAR PLATE

Tensile Strength	(ASTM-D-638)
#1	6,850 psi
#2	6,460 psi
#3	7,080 psi
Flexural Strength	(ASTM-D-638)
#1	10,700 psi
#2	9,870 psi
#3	9,130 psi
#4	9,140 psi
#5	10,440 psi
Shear Strength	(ASTM-D-732)
#1	4,030 psi
#2	4,020 psi
#3	3,800 psi
Hardness	(ASTM-D-785)
#1	112
#2	112
	113
#3	113
# 3 # 4	113

2.1.2 Material Composition

A number of plate properties are determined by the carbon-resin ratio in the plate. Of immediate interest for phosphoric acid stacks are specific conductance, contact (skin) resistance, and the stability in air and in phosphoric acid. These properties were tested over the composition range of 17 to 36% resin.

Test samples were molded in a flat mold from various mixtures of Colloid 8440 resin and graphite. A mixture of 2 high purity graphites was employed for all samples; it consisted of 11 parts by weight Asbury A99 synthetic (50µ average particle size) and 4 parts by weight Asbury 850 natural graphite (6µ average particle size). Molding conditions were the same as used for stack bipolar plate production, i.e., 4,300 psi at 350°F for 5 minutes. Initially, eleven samples were post cured for 6 hours at 400°F. Six samples were tested without post curing.

2.1.2.1 Specific Conductance

Resistivity measurements for the molded material samples were obtained by the conventional 4 point method. The values plotted in Figure 1 show that conductance varies linearly with concentration up to about 30% by weight resin; above this resin concentration, a rapid increase in resistance occurs. The resistivity range was found to be from 7 milliohm-cm at 17% resin to about 35 milliohm-cm at 33% resin. The test circuit for obtaining resistivity measurements for the sample graphite plates is shown in Figure 2. The data obtained for measuring the resistivity of the sample plates are shown in Table II.

2.1.2.2 Voltage Drop Thru Plate

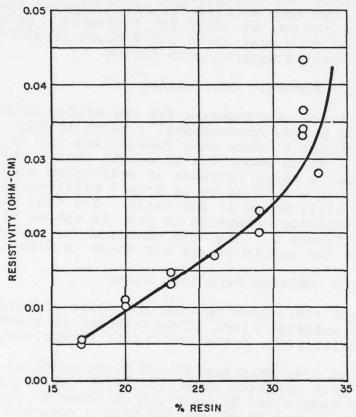
Contact resistance between the plate and electrode materials is of major interest since it contributes to stack resistance beyond the resistivity of the components themselves.

Voltage drop thru the molded plate material samples was determined with graphite paper (catalyst layer support) on either side of the sample and Ag contacts as shown in Figure 3 . The samples were tested at 80 psi and with a constant current of 2.75 amperes. The data of the test samples is shown in Table III. The apparent resistivity shown in Figure 4, has a value 5-10 times higher than the resistivity of the plate material. The difference between the two sets of figures is a measure of contact resistance between the electrode and the bipolar plate. It may be noted that contact resistance rises proportionately with material resistivity as resin content is increased. Also, a lowering of contact resistance was observed with increased post cure time. This result suggests that resin concentration on plate surface may be reduced during post cure; however, this needs to be verified in view of the difficulty of reproducing contact resistance measurements.

2.1.2.3 Density

Material density was determined by water immersion method. Density ranges from 1.92 g/cm 3 for 17% resin to 1.76 g/cm 3 for 33% resin as shown in Figure 5 . These data were taken on samples with a 6 hour, 400°F post cure. Table IV shows the data obtained for the individual test samples.

FIGURE I
BIPOLAR PLATE MATERIAL RESISTIVITY



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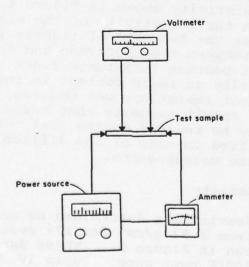


FIGURE 2
FOUR POINT RESISTIVITY MEASUREMENT

TABLE II

RESISTIVITY TEST DATA

- . 4 Point Measurement
- . 5" x 0.5 in. Sample
- . Voltage Measured @ 3" Length
- . 100 mA Input Current

SAMPLE NO.	% RESIN	THICKNESS (in)	VOLTAGE	RESISTANCE (ohms)	RESISTIVITY (ohm-cm)
4698	33	0.175	0.0376	0.376	0.0278
4686	32	0.148	0.0544	0.544	0.0341
4684	32	0.167	0.0587	0.587	0.0415
4683	32	0.142	0.0608	0.608	0.0365
4682	32	0.120	0.0649	0.649	0.0329
4690	29	0.151	0.0312	0.312	0.0199
4691	29	0.155	0.0354	0.354	0.0232
4688	26	0.151	0.0263	0.263	0.0168
4692	23	0.151	0.0201	0.201	0.0128
4693	23	0.149	0.0231	0.231	0.0146
4694	20	0.155	0.0150	0.150	0.0098
4695	20	0.152	0.0175	0.175	0.0113
4685	17	0.131	0.0088	0.088	0.0049
4697	17	0.155	0.0086	0.086	0.0056

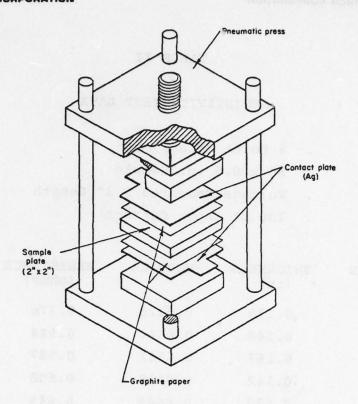
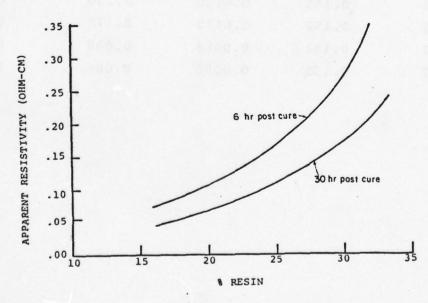


FIGURE 3.
CONTACT RESISTANCE SET-UP

D0506

FIGURE 4
BIPOLAR PLATE CONTACT RESISTANCE



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Table III

Resistivity Test Data

'2" x 2" Sample

'80 PSI

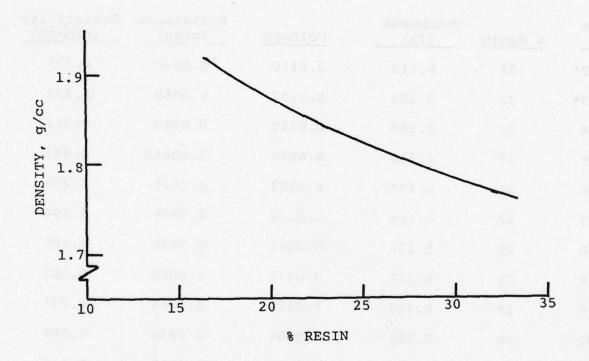
.70°F

'2.75A (100 ASF)

Sample No	% Resin	Thickness (In)	Voltage	Resistance (ohms)	Resistivity (ohm-cm)
4682*	32	0.113	0.0110	0.0040	0.359
4683*	32	0.152	0.0137	0.0050	0.334
4684	32	0.168	0.0165	0.0060	0.363
4685	17	0.116	0.0026	0.00095	0.083
4686	32	0.155	0.0123	0.0045	0.295
4687	32	0.164	0.0136	0.0049	0.296
4688	26	0.154	0.0084	0.0030	0.198
4689	26	0.152	0.0077	0.0028	0.187
4690	29	0.159	0.0121	0.0044	0.281
4691	29	0.161	0.0084	0.0030	0.189
4692	23	0.155	0.0048	0.0017	0.111
4693	23	0.142	0.0053	0.0019	0.136
4694	20	0.155	0.0043	0.0016	0.105
4695	20	0.147	0.0044	0.0016	0.111
4696	17	0.153	0.0025	0.00091	0.060
4697	17	0.157	0.0035	0.00127	0.082
4698	33	0.181	0.0135	0.0049	0.275

^{*}Horizontal hairline crack in sample.

FIGURE 5
BIPOLAR PLATE MATERIAL DENSITY



D0319

TABLE IV

Plate Density Determination

'Water Displacement Method

'2" x 2" Sample

'Analytical Balance

Sample No.	% Resin	Wt. in Air (Grms)	Wt. in H ₂ 0 (Grms)	Density (g/cc)
4682	32	12.7823	5.4103	1.73
4683	32	17.0091	7.0642	1.71
4684	32	19.4252	8.3563	1.75
4685	17	14.9926	7.2387	1.93
4686	32	18.3036	8.0178	1.78
4687	32	19.1111	8.2726	1.76
4688	26	18.8922	8.6934	1.85
4689	26	18.4688	8.4009	1.83
4690	29	19.0590	8.5904	1.82
4691	29	19.2300	8.5655	1.80
4692	23	19.1265	8.8838	1.86
4693	23	17.8511	8.1682	1.84
4694	20	19.4801	9.2213	1.90
4695	20	18.3108	8.4802	1.86
4696	17	19.7990	9.6825	1.96
4697	17	19.6788	9.3786	1.91
4698	33	21.1692	9.2198	1.77

2.1.2.4 Weight Change in Air

The bipolar plate material samples were exposed to air in an oven at 400°F, and weight changes were monitored periodically. The data plotted in Figure 6 shows a small weight loss proportional to the resin concentration. Where the same data is normalized with respect to the weight of resin in the plate, the weight loss can be seen to be independent of resin concentration.

2.1.2.5 Weight Change in H3PO4

The test samples were maintained in H_3PO_4 at 350°F for periods up to several months. The samples were periodically removed from the acid and washed in boiling H_2O until a neutral condition had been achieved (pH=7). The samples were then oven dried at 250 - 275°F for a period of 12 hours and weighed on an analytical balance. As seen in Figure 7, weight loss again is proportional to the concentration of resin in the plate. The total weight loss over a period of 5,000 hours was 1%, indicating increasing stability of the material with time as shown in Figure 8. No visual signs of attack of the samples by the acid were evident.

2.1.2.6 New Resins

Several new potential plate molding resins were examined during this phase of the project. This work was limited to preliminary compatibility tests with H₃PO₄.

Flat plates measuring 4" x 4" were molded with Kinel 5505 resin from Rhodia, Inc. The test samples molded at 460°F with 33% resin with the current graphite mix showed surface cracks after overweight immersion in $\rm H_3PO_4$ at 350°F.

An experimental Arofene resin from Ashland Oil Co. appeared suitable at 200°C, but needs to be tested in the full size molding process. The current resins of choice for bipolar plates are Arofene 890 and Colloid 8440. Most of the work on this project was carried out with the Colloid resin because of somewhat better molding characteristics.

2.1.3 Gas Distribution Pattern

Two modifications to the gas distribution pattern on the bipolar plate were evaluated on this project. The objectives for undertaking these modifications were

- (1) increased plate strength
- (2) improved moldability
- (3) larger cell active area

.FIGURE 6
BIPOLAR PLATE WEIGHT CHANGE IN AIR

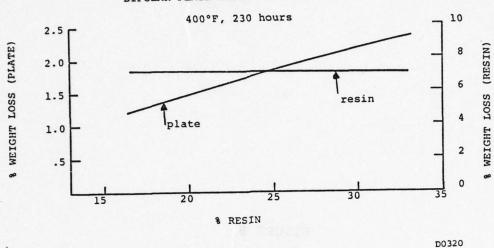
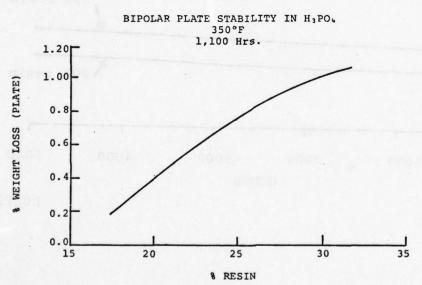
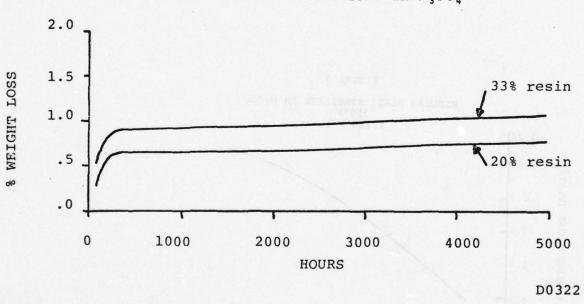


FIGURE 7



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FIGURE 8
BIPOLAR PLATE WEIGHT LOSS IN H₃PO₄



- (4) deeper electrode recess
- (5) lower tooling costs

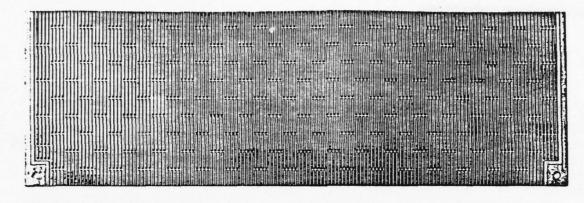
The bipolar plate designs are shown in Figure 9. Type B plate design has a staggered crossflow channel pattern, eliminating extended thin plate sections for greater plate strength. On the Type C plate, the perpendicular gas channels are eliminated, which greatly reduces tooling costs. A photograph of the plate pressing dies is shown in Figure 10.

Type B and C plates were designed with an electrode recess depth of .014 in. for the anode and 0.018 in. for the cathode compared to 0.012 in. for both anode and cathode on the earlier (Type A) plate. This change was undertaken to accommodate the increased electrode thickness resulting from the use of supported catalyst instead of platinum black on this project.

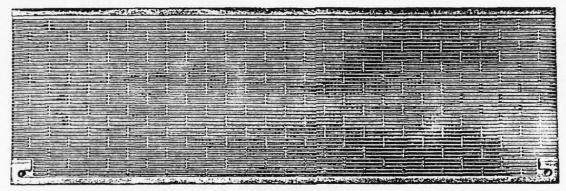
The presently available tooling in the 5×15 in size can produce the following plate configurations:

- A. Mold A: Present standard mold in use since the commencement of the MM&T program. This mold produces an enclosed electrolyte channel, electrode recesses .012 in deep, and straight line grooves perpendicular to the direction of the gas flow.
- B. Mold B: Modification of mold A as follows:
 - (1) Electrolyte channel not closed on top
 - (2) Electrode recesses 0.18 in (air side) and 0.14 in (fuel side) deep.
 - (3) Staggered grooves perpendicular to direction of gas flow for increased strength.
- C. Mold C: Modification of mold A as follows:
 - (1) No electrolyte channel mold is symmetrical on both air and fuel sides.
 - (2) Electrode recesses are same as for mold B.
 - (3) There are no grooves in the plate perpendicular to direction of gas flow.

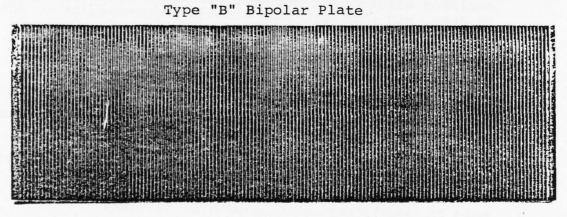
Initial stack performance was not affected by gas distribution pattern design. This is expected since both fuel and air field depth as well as overall plate thickness remain unchanged. However, the deeper electrode recesses in B and C plates have improved edge seal gastightness of the stacks.



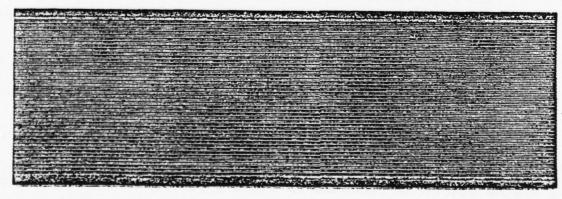
Air Side



Fuel Side

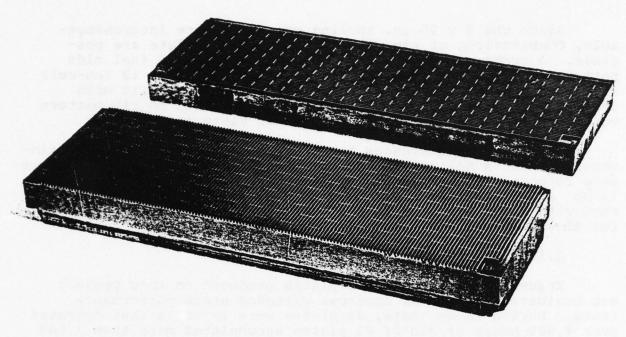


Air Side

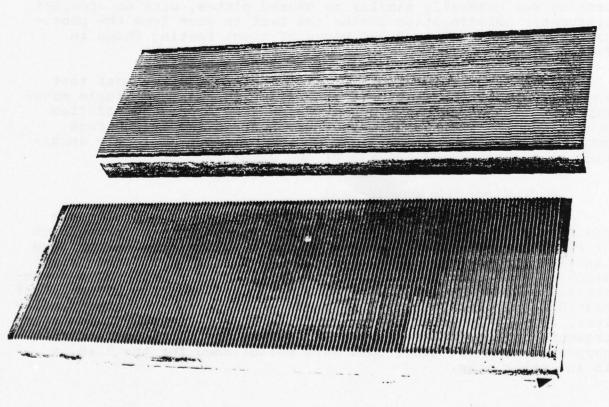


Fuel Side

Type "C" Bipolar Plate
FIGURE 9
BIPOLAR PLATE DESIGN



Type "B" Plate Mold



Type "C" Plate Mold

FIGURE 10
PLATE PRESSING DIES

Since the 5 x 15 in. tooling components are interchangeable, combinations of rib patterns on the same plate are possible. A number of plates were molded with the "A" fuel side pattern combined with the "B" air pattern. The last 10 ten-cell stacks as well as the 80-cell (2.1 kW) stack were built with these plates. Also, some plates were molded with no rib pattern on one side for use as the first or last plate in the stack.

Dimensional tolerances for the plates were monitored continuously. Plates not conforming to a ±0.002 in. thickness tolerance were rejected. For standard molding procedures the rejection rate is no worse than 20%. Plates were also checked 100% for gastightness with 2 psi hydrogen as the test gas. The rejection rate for this test was less than 4%.

2.1.4 Endurace in Stack Tests

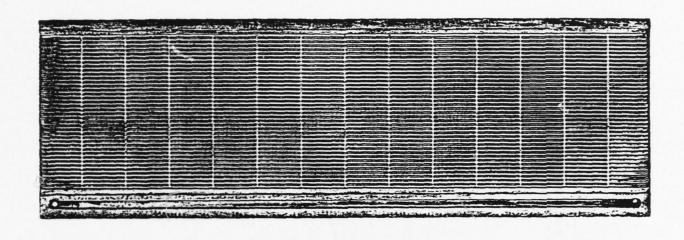
Endurance of the bipolar plates produced on this project was evaluated as part of numerous extended stack performance tests. During these tests, 24 plates were in cells that operated over 4,000 hours at 350°F; 62 plates accumulated more than 3,000 hours, and 106 plates were in stacks which were tested for over 2,000 hours. The appearance of the plates following endurance testing was generally similar to unused plates, with no apparent systematic deterioration during the test as seen from the photograph of a plate after 7,000 hours of stack testing shown in Figure 11.

Soft areas were observed on some plates during post test analysis. This condition was generally limited to a single manufacturing run of about 20 plates which were molded at modified conditions (20 sec. vs 5-10 sec. pre heat in the mold before compression). We attribute the softening to incompletely densified plate sections (porostiy of the plate).

2.2 Electrode Development

As part of an earlier effort toward development of stack manufacturing process (1), ERC evolved a technique for production of fuel cell electrodes using platinum black or rhodium-platinum catalyst for portable powerplant (5 in. x 15 in. electrode size) stacks. Other work at ERC with small (2 in. x 2 in.) cells demonstrated performance gains and catalyst cost reduction potential of platinum-on-carbon compared to platinum black as the cathode catalyst, as seen from the polarization curves of Figure 12. On the present project, manufacturing techniques for platinum-on-carbon supported catalyst electrodes were established for the 5 in. x 15 in. stack size.

⁽¹⁾ Contract DAAK02-74-C-0367, Final Report, 1977



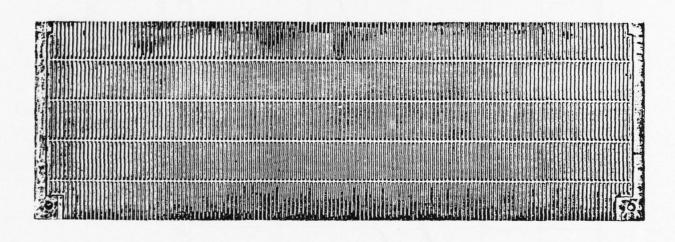
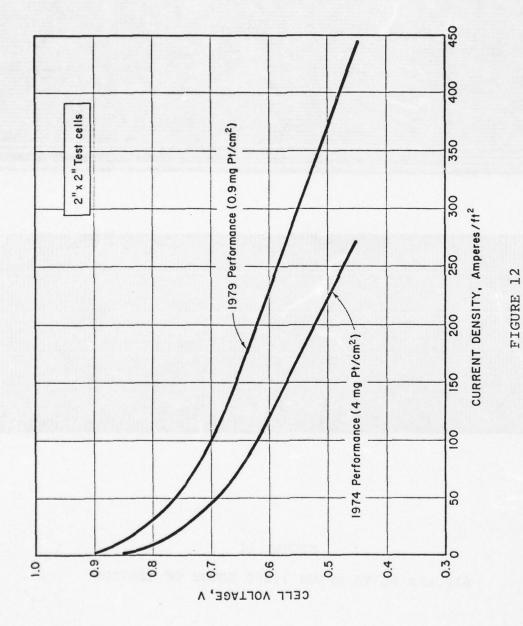


FIGURE 11
BIPOLAR PLATE AFTER 7,000 HOURS OF TESTING



SMALL CELL PERFORMANCE

DO503

The electrode manufacturing process is summarized in Figure 13. The process was employed to produce electrodes with platinum loading ranging from about 0.2 to 1.0 grams/ft². Physical characteristics of electrodes having 0.6 and 0.9 grams Pt/sq. ft. of electrode are listed in Table V. Electrodes with these catalyst loadings (0.6 anode, 0.9 cathode) were used in most stacks in the last phase of the project, when ten 10-cell stacks for reliability evaluation and the 2.1 kW (80-cell) stack for delivery to MERADCOM were built. No rhodium or tungsten oxide is used in the anodes.

Performance of 5 in. x 15 in. cell stacks built with the supported platinum catalyst electrodes was higher than with platinum black catalyst. Individual cell voltages in some stacks were 0.65-0.67V at 100 ASF compared to 0.61-0.62 obtained with Pt black electrodes with 2g Pt/cm² catalyst loading.

Stacks built with anodes having 0.3-0.6g Pt/ft² were tolerant to 1% CO in the fuel. Detailed performance data for stacks built with the supported catalyst electrodes are shown in the stack test section (Section 4.0) of this report.

2.3 SiC Matrix Development

Silicon carbide is among the very few materials which appear to be completely inert to phosphoric acid at fuel cell operating temperatures. Fuel cell matrices made from fine grit SiC powder as well as from SiC whiskers have been evaluated at ERC in small (2 in. x 2 in.) cell test rigs. As a separate task on this project, matrix manufacturing and stack assembly techniques were developed to adapt the SiC matrix process to the 5 in. x 15 in. stack hardware.

Although SiC matrices have been successfully prepared in the past by the papermaking $\operatorname{process}^{(1)}$, the unavailability of a consistent quality SiC whisker material (obtained previously from Exxon Enterprises, Inc.) limited the material choice to SiC powder. Throughout this project, 1000-grit SiC powder from Carborundum Corp. was employed.

The matrices were prepared by applying an aqueous SiC slurry formulated as shown in Table VI directly onto the electrode by means of the coating machine shown in Figure 14. The electrode is placed on the bed of the machine and a uniform layer of the slurry is spread onto the electrode by a wire-bound bar mounted in a yoke driven by variable speed motor. The thickness of the matrix is controlled by the gauge of the wire on the casting bar and also by the clearance between the bar and the electrode surface. After casting, the matrix is air dried and sintered at 275°C. The resulting matrix typically has a thickness of 0.007 in. and porosity in the range 50-60%. Bubble pressure for the SiC matrix prepared by this process was somewhat higher than bubble pressure measured for Kynol matrices (15-20 psi vs ~10 psi).

⁽¹⁾ Contract DAAK02-75-C-0045, Final Report

Page No.

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NOTES:

- 3800
- SHELL SOL GUARD ALL CO.
 AMMONIUM BICARBONATE ANALYTICAL REAGENT, FISHER SCIENTIFIC CO.
 TFE DUPONT
 FEP DUPONT

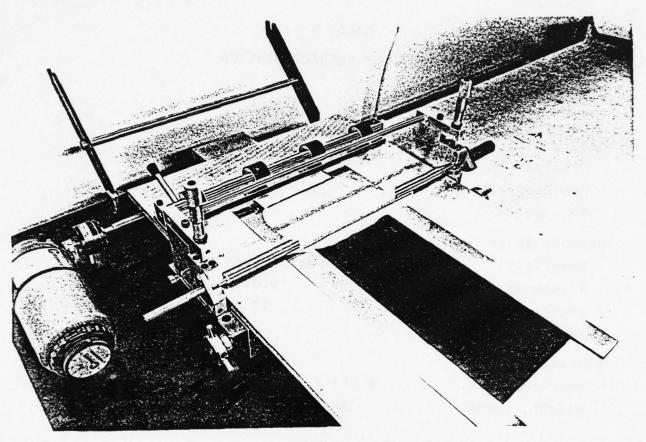
TABLE V
ELECTRODE CHARACTERISTICS

Catalyst layer	Cathode	Anode
Catalyst	10% Platinum on	Carbon
Loading, gPt/ft ²	0.9	0.6
TFE, wt %	40	40
Support layer		
Material*	Graphite paper,	83% porous
Thickness, in.	0.017 ±	.001
Weight, g/ft ²	13.0 ±	.13
FEP, wt %	20	
Electrode		
Thickness, in.	0.023 ± .002	.0021 ± .002
Weight, grams	18 ± 2	13 ± 2

^{*} Supplied by Stackpole Carbon Co.

TABLE VI SiC MATRIX

Material	w/o, dry basis
Silicon Carbide (1000 grit)	96 - 98
Polyethylene Oxide	0.3
Polytetrafluoroethylene (TFE 30)	2 - 4



P0095

-

FIGURE 14
COATING MACHINE

The bubble pressure of matrices were measured with the testing apparatus shown in Figure 15. The prewet coated electrode was placed matrix down in the appropriate area of the bottom stainless steel plate. The top plate was then properly secured over the sample. Water was placed in the water retention area and $\rm H_2$ pressure was slowly increased. The pressure was constantly monitored by the pressure gauge and by the manometer. The pressure point when bubbles were observed through the water layer on top of the matrix was determined.

Several stacks utilizing the SiC matrix were constructed and tested. Performance of these stacks was generally comparable to that obtained with stacks having Kynol matrices, except for open circuit voltage which was somewhat lower for the SiC stacks. This may have been due to a modified stack assembly technique used with the SiC matrix. Stack construction details and stack test results are presented elsewhere in this report (Sections 3.0 and 4.0, respectively).

3.0 STACK ASSEMBLY DEVELOPMENT

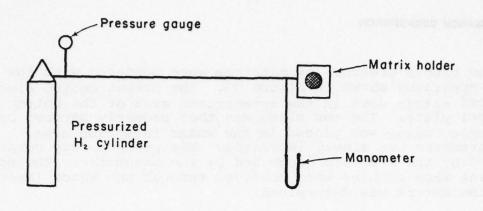
Effective separation of the gases in the stack and gastight manifolding of the fuel are essential for high stack performance and fuel utilization. Methods for achieving gastight assemblies were studied on this project.

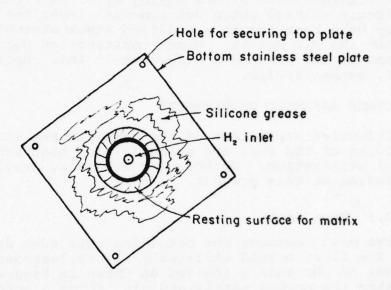
3.1 Edge Seals

Two basic methods for obtaining cell edge seals were evaluated. The first method utilizes a fluoroelastomer cement (1) around the edges of the matrix applied as shown in Figure 16. The cement penetrates the porous matrix and also forms a bond to the bipolar plate as shown in Figure 17. The stack is assembled with dry components, the electrolyte being allowed to wick into the matrix from the electrolyte fill channel.

The second method depends entirely on H₃PO₄ to provide edge sealing, with no cement being used (except to position the electrodes on the bipolar plate). The stack is assembled with prefilled matrices, which reduces the time required for wicking of the stack via the electrolyte fill channel to 1-2 days from 4-5 days. The electrolyte fill channel can be seen in the cutaway stack section drawing shown in Figure 18. Acid of 98% concentration is used in the wet assembly technique. Prior to use the acid is heated to 170°F. A syringe is used to apply 16 ml of acid uniformly over the 5" x 15" matrix.

⁽¹⁾ C-328 Viton RTV Cement, The Connecticut Hard Rubber Co.





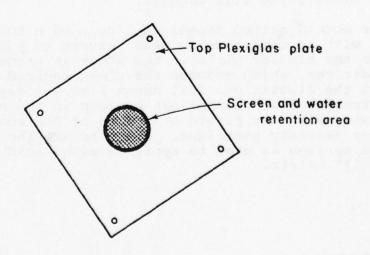


FIGURE 15
BUBBLE PRESSURE TESTING SET-UP

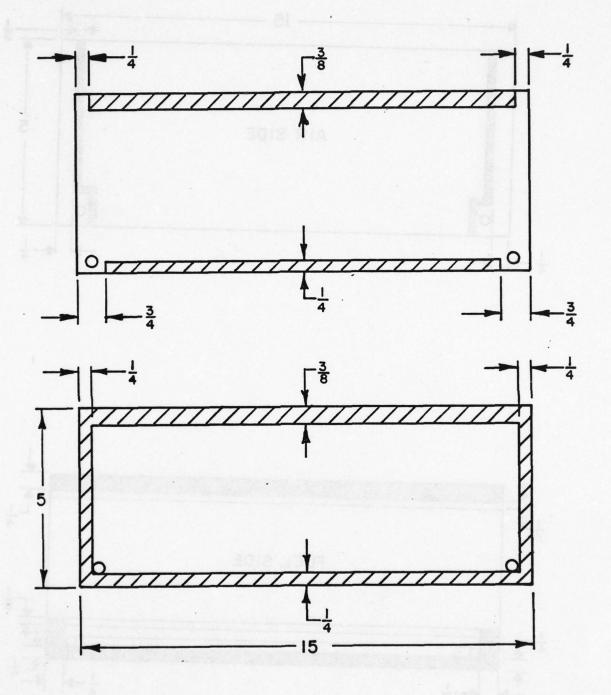
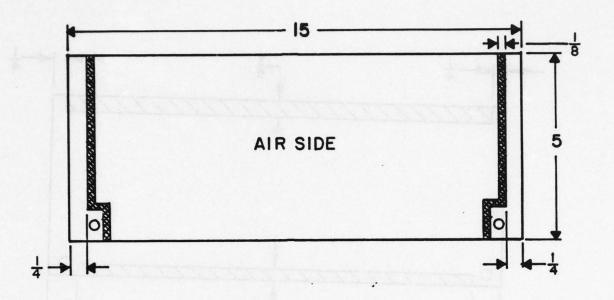


FIGURE 16.
CEMENTED AREA ON MATRIX



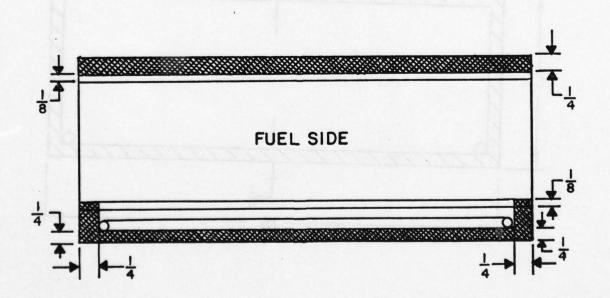
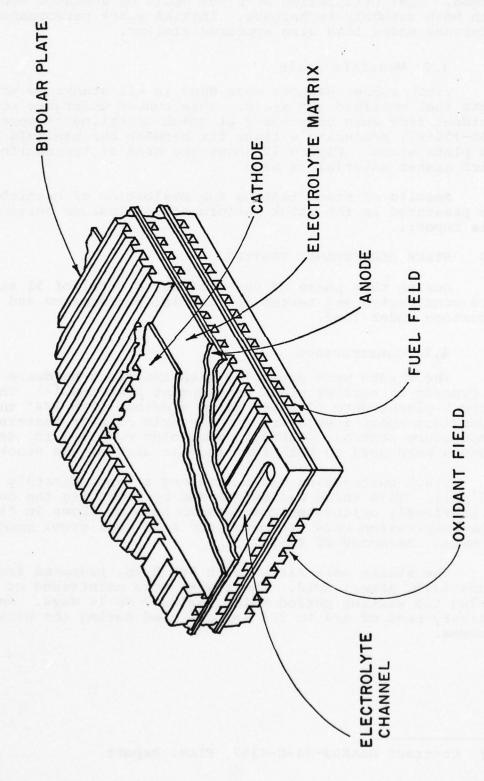


FIGURE 17.
CEMENTED AREA ON PLATE

STACK CUTAWAY

FIGURE 18



27

Testing of 3- and 10-cell stacks assembled by both methods did not indicate superior performance or endurance for either method. Fuel utilization over 90% could be achieved consistently with both assembly techniques. Initial stack performance and endurance under load also appeared similar.

3.2 Manifold Seals

Viton rubber gaskets were used in all stacks to achieve tight fuel manifold gas seals. This rubber undergoes some residual flow when compressed at stack operating temperatures (250-350°F), producing a tight fit between the manifold and the plate stack. Figure 19 shows the area of the manifold over which gasket material is used.

Results of stack testing for evaluation of gastightness are presented in the stack performance evaluation section of this report.

4.0 STACK PERFORMANCE TESTING

During this phase of the project, a total of 51 stacks were constructed and tested for initial performance and for endurance under load.

4.1 Construction

The stacks were assembled with the stack hardware described in reports on earlier stack development programs. (1) The end support plates were 1" thick solid aluminum with 1/4" thick fiberglass-epoxy insulators and electric blanket heaters for temperature control. Phenolic manifolds with 1/2 in. deep plenums were used on both fuel and air side of the stack.

Stack compression was maintained at approximately 5400 lbs (72 psi). This value was determined by measuring the deflection of previously calibrated compression bars as shown in Figure 20 (The compression bars are made from AISI tool steel hardened to Rockwell hardness of 46-48).

The stacks were filled with 98% $\rm H_3PO_4$ prepared from Reagent Grade (J.T. Baker) acid. The stacks were maintained at 250°F during the wicking period which lasted 7 to 10 days. An acid delivery head of 1/2 to 3/4 in. was used during the wicking process.

⁽¹⁾ Contract DAAK02-74-C-0367, Final Report

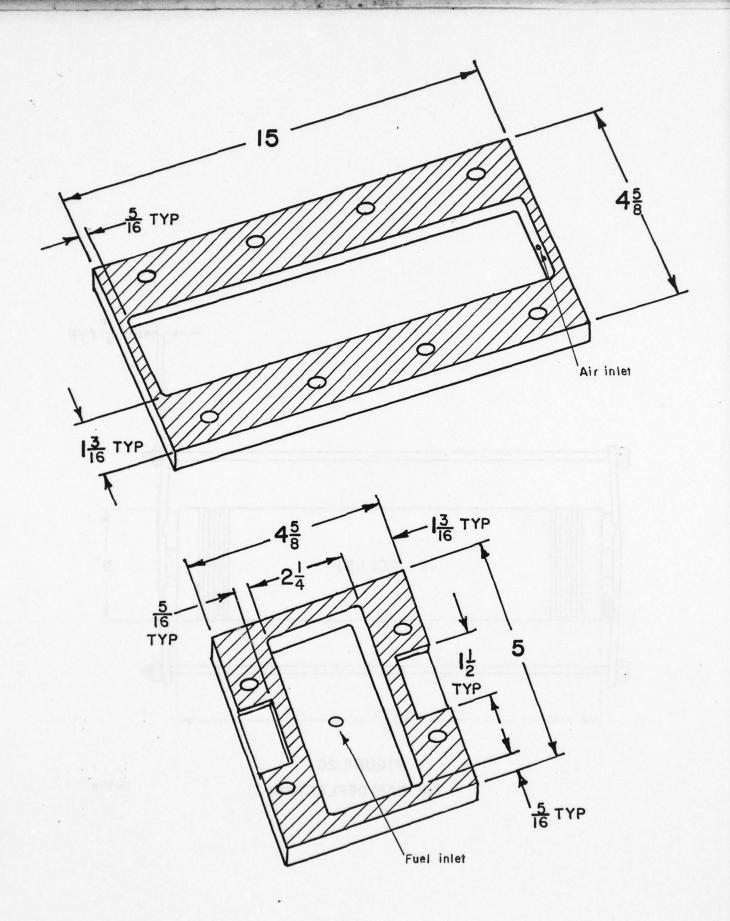
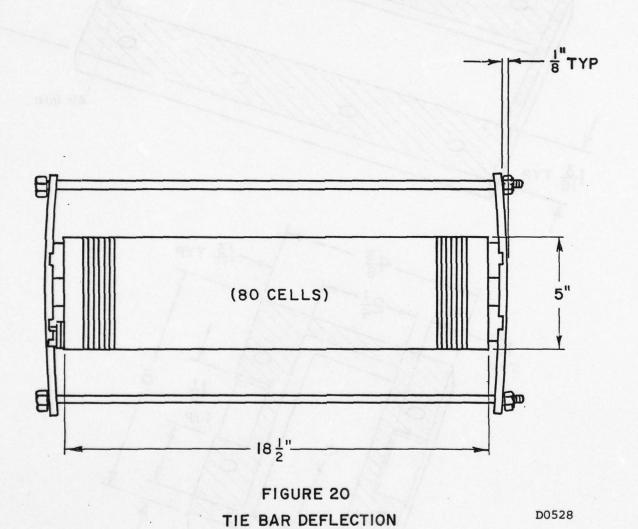


FIGURE 19

D0527

MANIFOLD SEALS



4.2 Test Procedure

Stacks were evaluated for initial performance under a number of operating conditions and for performance stability under continuous load conitions. Most of the testing was done with hydrogen fuel, but some tests were also conducted with simulated reformed fuel (SRF). Air rates thru the stacks were maintained at 6 to 10 times of stoichiometric reaction requirements. Diagnostic tests with oxygen as the cathode gas were conducted to periodically monitor cathode performance. A test set-up schematic is shown in Figure 21.

Stack temperatures were controlled by the endplate heater blankets and by air temperature and flow rate. All operating parameters (temperature, current, voltage, flow rates) were monitored on the test panels shown in Figure 22. A facility for simultaneous performance testing of twelve stacks was assembled for the purposes of this project.

4.2.1

The temperature profile of the ten cell stack No. 1050 was investigated by inserting thermocouples into the air channels at 1 inch intervals. The air inlet temperature was 250°F and the $\rm H_2$ inlet temperature was 75°F. The air supplied to the stack was a constant 58 liters/minute and the $\rm H_2$ was a steady 3.4 liters/minute while operating at 40A.

As expected, the temperature was found to be lower near the air and fuel inlets. In all instances the hottest point in the long direction of the stack was found to be near the center. In the vertical direction the hottest point was approximately 2 inches from the air outlet. The first and last two cells also operate slightly cooler than the center cells. A temperature profile for the sixth cell from Stack 1050 is shown in Figure 23.

4.3 Three-Cell Stack Tests

Three-cell stacks were assembled to test both component performance and assembly endurance. The three-cell stacks were also used to evaluate effect of several operating parameters on cell performance since, unlike 10-cell stacks, these stacks showed relatively small temperature variation cell to cell and inlet to outlet due to the proximity of endplate heaters to all cells. The major construction variables and test results for these stacks are summarized in Table VII.

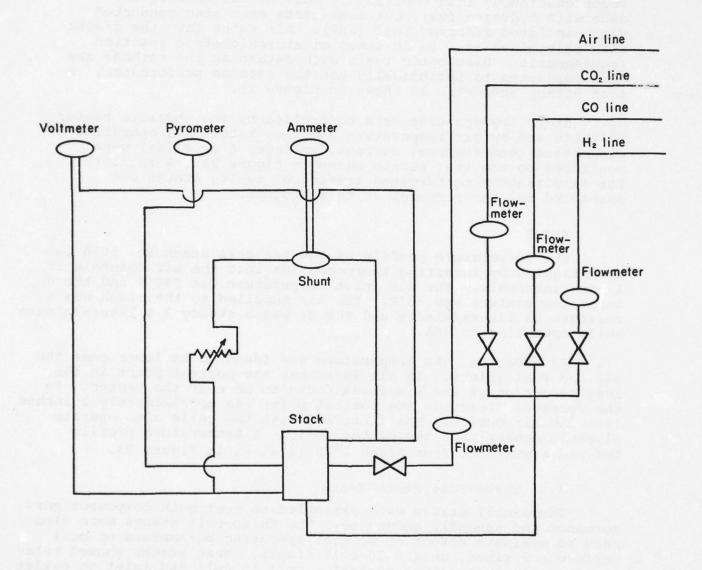
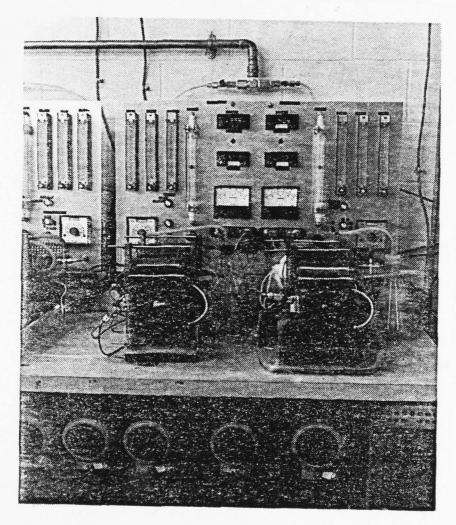


FIGURE 21

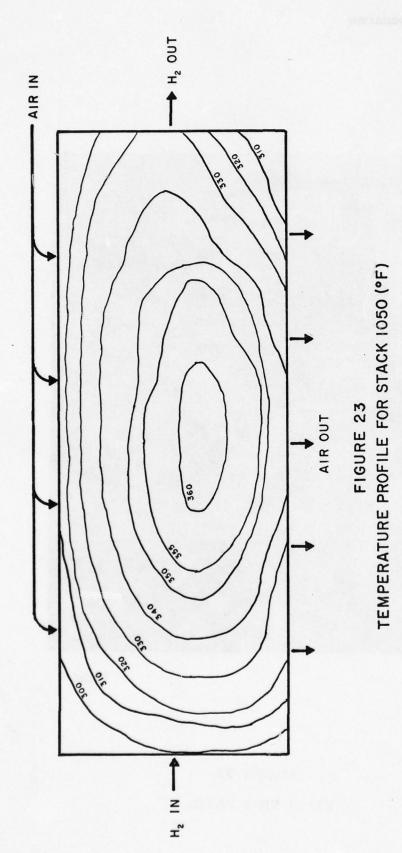
TEST SET-UP SCHEMATIC

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P0096

FIGURE 22 STACK TEST PANEL



SUMMARY
DATA
STACK
THREE-CELL
VII
TABLE

NO. TYPE NO. TYPE NO. TYPE NO. TYPE NO. TYPE NO. NA. NA. NA. NA. NA. NA. NA. NA. NA. NA	MATRIX K K K K K K K K K K K K K K K K K K K	ANODE CATALYST TYPE 9/18 PB 4.2 Pt/C 3.4 PB 3.3 PB 3.3 PB 3.6 PB 5.6 PB 5.6 PB 5.6 PB 5.6 PF/C 6.6	7 Ft.2 4.2 3.4 3.2 3.3	CATALYST TYPE 9/	YST 9/Ft	ASS'Y	CET	INITIAL 3	AGE, mV	l (a	AVERAGE	3	HOURS	REMARKS
TYPE AA	ATRIX K K K K K K K K K K K K K	TYPE PB PB PB PB PB PB PB PB PC	9/Ft² 4.2 3.4 3.2 3.3	TYPE	g/Ft	ASS'Y	1	2	3	1	2		TESTED	REMARKS
900,0 Mayor 58,326+ s	ж ж к к к к ; ; ; ; ; ; ; ; ; ; ; ; ; ;	PB PB PB PB PB PB PC Pt/C	3.3.2.4	2				-	1	1	-			
Web although balant s	S	Pt/C PB PB PB PB PC Pt/C	3.2	PB	.36	۵	540	540	520	440	440	460	7.0	
Callevon Dajzet s	N	PB PB PB PB PB Pt/C	3.2	Pt/C	.63	Q	200	210	530	440	200	475	10	g ide
AMERICA DELIES 1 :	N	PB PB PB PB Pt/C	3.3	Pt/C	96.	0	570	570	570	525	545	530	1180	
toyog Dalizat s	х х х х х х х х х х х х х х х х х х х	PB PB PB PB Pt/C Pt/C	20	Pt/C	.85	Q	620	630	630	570	280	580	2820	SRF Testing (100 Hours)
906 063864 6	Si X X X X X X	PB PB PB Pt/C Pt/C	2.0	PB	2.9	M	620	630	620	570	290	580	2700	SRF Testing (700 Hours)
0.081264	Sic	PB PB Pt/C Pt/C	3.4	PB	2.8	Q	570	290	580	535	550	525	40	
28.189 t	* * * * * *	PB Pt/C Pt/C	3.0	Pt/C	.67	Q	520	260	570	465	485	485	1130	SRF Testing (100 Hours)
Jeef s	* * * * *	Pt/C Pt/C	3.5	PB	3.0	M	630	019	620	575	570	575	2630	20 V 10 V
AA AA	* * * *	Pt/C	.84	Pt/C	.84	Q	620	620	620	575	580	580	6850	SRF Testing (300 Hours)
AA	× × ×		86.	Pt/C	66.	Q	490	530	440	465	200	420	330	
	× ×	Pt/C	1.0	Pt/C	66.	Ω	490	530	520	420	475	460	260	
AA	×	Pt/C	66.	Pt/C	1.0	Q	540	550	490	465	505	450	260	
16 AA		Pt/C	1.0	Pt/C	.95	M	450	540	470	425	520	450	270	200
W	Ж	Pt/C	.85	Pt/C	.91	Q	019	630	490	570	570	420	1080	
18 CC	×	Pt/C	.65	Pt/C	. 80	M	,	'	1	,	1	•	0	Internal Short
19 AB	K	PB	2.4	Pt/C	. 85	W	630	640	620	280	290	540	1900	
20 AA	×	PB	2.8	Pt/C	.80	Q	620	099	630	605	615	595	510	Submitted to MERADCOM
21 AA	М	PB	2.6	Pt/C	87	D	550	009	009	515	585	585	835	
22 AB	×	PB	3.2	Pt/C	.72	M	009	029	620	580	605	575	40	
23 BB	×	Pt/C	.23	Pt/C	.51	Q	510	009	290	465	575	575	255	
BB	×	Pt/C	.30	Pt/C	. 58	×	550	390	490	550	390	490	09	
BB	Ж	Pt/C	.38	Pt/C	.62	M	620	630	630	595	585	595	5220	
26 CC	Ж	Pt/C	89.	Pt/C	17.	M	620	620	620	019	009	019	10	
AB	×	Pt/C	19.	Pt/C	.81	۵	620	640	640	615	625	620	1100	Submitted to MERADCOM
28 AB	M	Pt/C	.61	Pt/C	.82	M	099	099	640	640	029	640	1350	
30 CC	×	Pt/C	.94	Pt/C	88	M	630	009	630	019	210	585	250	
BB	X	Pt/C	.59	Pt/C	.84	Q	630	029	059	595	630	630	4490	
32 AB	×	Pt/C	.62	Pt/C	.81	Q	099	059	029	620	019	605	4010	SRF Testing (408 Hours)
AB	Ж	Pt/C	.57	Pt/C	.77	W	640	059	620	019	625	575	4270	SRF Testing (2165 Hours)
AB	Ж	Pt/C	.62	Pt/C	.87	۵	640	099	099	615	635	625	3720	T I I I I I I I I I I I I I I I I I I I
AB	×	Pt/C	69.	Pt/C	69.	M	350	400	390	350	400	370	150	8 6 6 8
39 AA	Sic	Pt/C	06.	Pt/C	06.	Q	640	620.	1	620	019	1	4005	Two-Cell Stack
52 AB	Sic	Pt/C	.58	Pt/C	06.	۵	009	640	620	585	630	019	1130	Submitted to MERADCOM
53 AB	Sic	Pt/C	.54	Pt/C	.55	Q	630	640	019	625	635	009	840	
54 AB	Sic	Pt/c	.53	Pt/C	.63	0	620	630	620	019	625	019	310	Submitted to MERADCOM
Plate AA - (A) Furl AB - (A) Fuel	Side, (A	(A) Air Side (B) Air Side	le Catalyst	lyst PB Pt/C	1 1	Platinum Rlack 10% Platinum, 90% Carbon	8 Carbon		Matrix K SiC	1.1	Kynol Silicon Carbide	bide	2.	SRF - Simulated Reformer Fuel 74% Hz, 25% CO2, 1% CO
BB - (B) Fuel	Side,	(B) Air Side	0					Ass'y	٠,۲ D	- Dry 7	Assembly	Dry Assembly		

.

4.3.1 Initial Performance

In general, peak cell voltages were observed within a few days from placing the stack on test. However, a few stacks did show an increase of 30-40 mV/cell after they had operated at 40A and 350°F for 1,000-1,500 hours. All of these stacks had supported catalyst cathodes, and the operating voltage increase probably indicates continued wetting of the catalyst with the electrolyte.

The highest operating potentials in this series of stacks were obtained with Kynol matrices and supported catalyst electrodes with anode and cathode loadings around 0.6 and 0.9 Pt/ft², respectively. There was no apparent difference in performance between stacks assembled with wet and dry matrices. There was also no significant difference between stacks built with the various plate groove patterns. Polarization data for some of these stacks is shown in Figure 24.

4.3.2 Carbon Monoxide Tolerance

Effect of CO in the fuel was evaluated for Stack 37 over the temperature range of 265 to 355°F. Hydrogen utilization for these tests was 80%. As seen from the data plotted in Figure 25, the anodes become progressively more CO tolerant with increasing temperature. At 315-320°F, the effect of 1.7% CO in the inlet is less than 10 mV at a current density of 100 ASF and about 5 mV at 350-355°F, the projected operating temperature for this stack design.

4.3.3 Performance with SRF

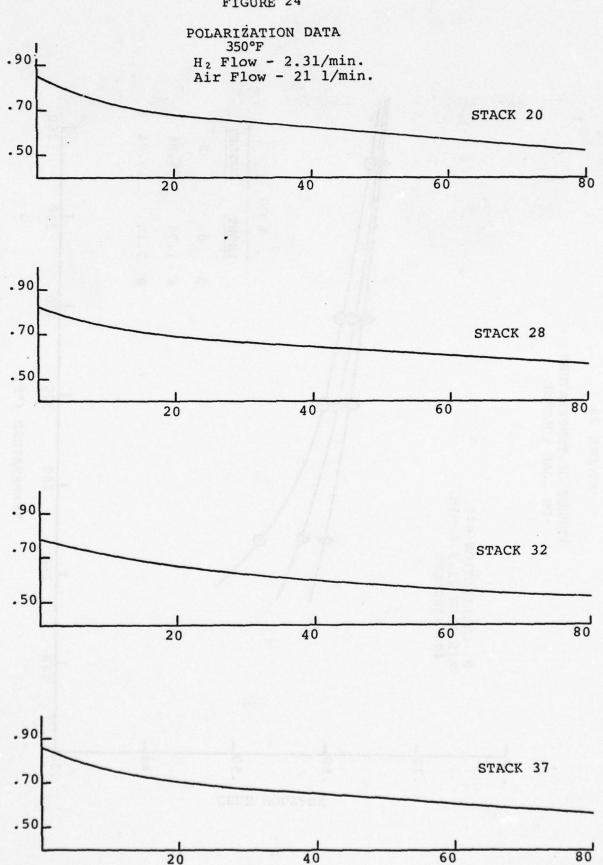
Several stacks were operated continuously on simulated reformed fuel (SRF) containing 72% $\rm H_2$, 24% $\rm CO_2$, 1% $\rm CO$, and 3% $\rm H_2O$. Cell operating voltages were recorded periodically for the operating condition of 100 ASF at 350°F while operating on SRF and on pure hydrogen. Results of this testing are shown in Table VIII for Stack 34. The hydrogen gain for this stack remained typically around 15-20 mV/cell throughout the duration of the test (over 2,000 hours).

4.3.4 Effect of CO2

The effect of CO_2 added to the fuel on cell voltage can be seen in Figure 26. The results are consistent with the voltage effect observed with SRF.

4.3.5 Performance Stability

Selected stacks were continued on test at 100 ASF at 350°F to observe performance stability. Six stacks were tested for more than 4,000 hours; another 4 stacks were tested beyond 3,000 hours.



Load Current, Amperes

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AVERAGE CELL VOLTAGE

FIGURE 25

EFFECT OF TEMPERATURE ON LOAD VOLTAGE

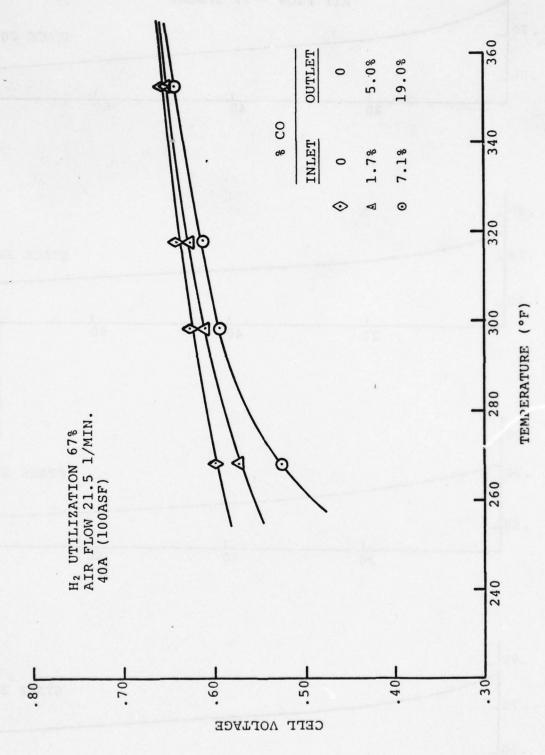


TABLE VIII

PERFORMANCE WITH SRF *

40A, 340-360F

80% Fuel Utilization

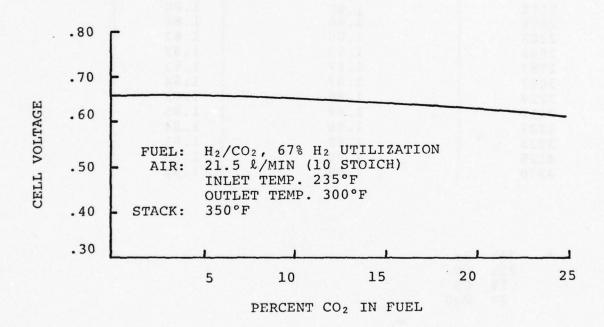
TIME	3-CELL STACK VO	
(HOURS)	Н 2	SRF
		1
2118	1.85	1.80
2259	1.86	1.82
2403	1.86	1 82
2595	1.87	1.82
2787	1.87	1.82
3027	1.87	1.84
3219	1.86	1.83
3411	1.88	1.85
3721	1.88	1.84
3933	1.88	1.83
4125	1.88	1.85
4270	1.88	1.82

73% H₂ 23% CO₂

1% CO

3% H₂O

FIGURE 26
EFFECT OF CO₂ ON LOAD VOLTAGE



Some of these stacks were terminated voluntarily at the end of the first phase of the project. Other stacks were terminated because of poor fuel utilization or low operating voltage.

Oxygen gain was determined periodically on stacks undergoing endurance testing. In general, oxygen gains remained in the 60-80 mV range at 100 ASF, indicating cathode catalyst layer stability. This can also be seen from the voltage-time curves plotted in Figure 27.

4.3.6 Temperature Cycling

Temperature cycling was performed on Stack 1047. The cycle consisted of a 2 hour shutdown and 6 hours of operation. During shutdown, the stack cooled to 140-150°F. At this point the stack and fuel heaters were activated, the fuel turned on, and the load bank connected. The warmup curve for the stack obtained under these conditions is shown in Figure 28.

Under these test conditions 120 cycles were completed. The load voltage remained stable but there was a slight decline in the open circuit voltage over the cycling period. Some acid loss was observed in the form of drops of acid on the bottom of the stack. This acid loss was probably caused by the low start temperature of the stack and the long operating time below 250°F (~ 10 min). Higher start temperatures and faster warmup rates will be used in future tests. Also, low heat capacity endplates will be used to allow a more uniform temperature profile in the stack.

4.3.7 Electrolyte Replenishment

Replenishment of electrolyte in the stacks is possible thru the filling tubes at any time during operation or storage (with plates in the vertical position). During the extended testing of stacks in this project, acid was added at irregular intervals by wicking for 2-4 days, usually after a reduction of open circuit or load voltage was observed. The actual interval for needed electrolyte additions was not determined on this project, but frequency of acid additions generally varied between 1,500 and 3,000 hours of stack operation.

4.4 Ten-Cell Stack Tests

Fifteen ten-cell stacks were assembled as described in Section 4.1. The initial five builds were used to verify designs and assembly procedures developed as part of the 3-cell stack construction task. Ten additional 10-cell stacks were dedicated to evaluation of reliability of the two assembly procedures, i.e., the "dry" assembly using Viton rubber cement and the "wet" assembly using prefilled Kynol matrices. The stacks were assembled with electrodes utilizing 10% Pt on carbon catalyst, 0.85g Pt/ft² for cathode, and 0.6g Pt/ft² for anode. The standard Kynol matrices

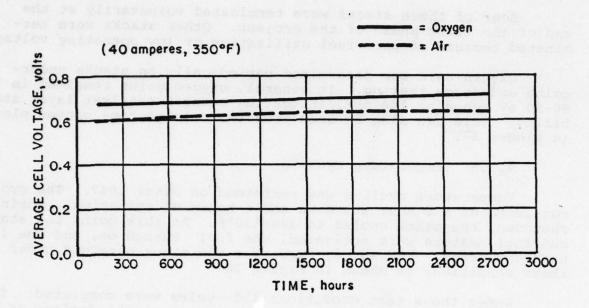


FIGURE 27
CELL VOLTAGE STABILITY

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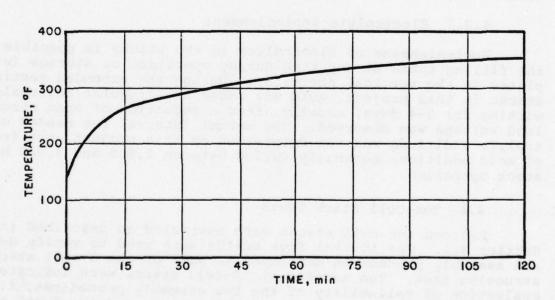


FIGURE 28
IO-CELL STACK WARM-UP

D0516

were employed, and bipolars had "A" pattern on the fuel side and "B" pattern on the air side. Five of the ten stacks were built by the "dry" assembly method, and the remaining five by the "wet" method as described in Section 3.1.

Testing of these stacks was conducted in the usual fashion, i.e., measurements were made of the open circuit voltage, load voltage at 25 to 200 ASF, fuel utilization, performance on hydrogen and SRF, and performance stability under continuous load at 100 ASF.

All of the 10-cell stack construction and test data are summarized in Table IX. For the last 10 ten-cell stacks built on this phase of the project, cell open circuit and load potentials (100 ASF) are listed in Tables X and XI, respectively. The data show uniform performance for individual cells in a stack as well as between stacks. (The somewhat lower cell potentials at the ends of the stack are associated with lower cell temperatures.) Polarization curves for these stacks are plotted in Figures 29 and 30. Two stacks were operated on SRF; results of these tests are shown in Table XII. The above results indicate no difference in initial performance between the two methods of stack assembly.

Endurance testing of all stacks was conducted at 100 ASF and 350°F on hydrogen fuel. No stack failures were encountered during 500 hours of operation, the duration of initial stack qualification according to test plan. Voltage-time plots for these tests are shown in Figures 31 and 32.

Several 10-cell stacks were continued on test beyond the initial 500-hour test period. Operating on hydrogen at 350°F and 100 ASF, performance remained stable for over 3,000 hours of testing as shown in Figure 33.

4.5 2.1 kW Stack

An 80-cell stack, nominally rated at 2.1 kW, was constructed and tested. This stack was assembled with Viton rubber cement seals (dry assembly), and the electrolyte was allowed to wick into the matrices in the usual fashion. Specifications for the components used to build this stack and stack component weights are listed below in Tables XIII and XIV, respectively.

Gas manifolds, blower, and recirculation ducts of the type employed previously for 2 kW stacks $^{(1)}$ were fabricated and installed. A 2 kW nichrome wire electric heater was installed in the recirculating duct for startup heat as shown in Figure 34.

⁽¹⁾ Contract DAAG53-76-C-0118, Final Report

TABLE IX TEN-CELL STACK DATA SUMMARY

STACK	PLATE	ANODE	CATALYST		AVERAGE CELL VOLTAGE mV @ 40A, 350°F	LL VOLTAGE	HOURS	a la
NO.	TYPE	g/Ft2	g/Ft2	ASS'Y	INITIAL	AVERAGE	TESTED	REMARKS
10	AA	.80	. 80	Q	630	590	3220	27 80 8V 10 10 10 10 10 10 10 10 10 10 10 10 10
29	AB	.39	56.	D	610	909	1305	Submitted to MERADCOM
33	AB	.65	98.	M	630	009	365	
35	BB	.61	,81	M	620	009	1535	
36	BB	.62	.81	M	. 029	620	09	
40	AB	.61	. 88	W	909	585	550	
41	AB	.58	68.	M	595	290	1185	
42	AB	.61	.80	M	009	595	1355	Submitted to MERADCOM SRF Testing (38 Hours)
43	AB	.63	.85	Z	595	580	2795	
44	AB	09.	.84	W	290	575	1385	
46	AB	09.	. 89	D	009	595	670	<pre></pre>
47	AB	.61	68.	Q	595	575	2020	Temperature Cycling (121 ON-OFF CYCLES)
48	AB	.58	.87	D	610	595	2005	
49	AB	09.	68.	D	610	580	2310	9 .00 lb a lb
50	AB	.55	.84	Q	595	575	2220	Horizontal Operation for 1800 Hours

Plate AA - (A) Fuel Side, (A) Air Side

AB - (A) Fuel Side, (B) Air Side

BB - (B) Fuel Side, (B) Air Side

BB - (B) Fuel Side, (B) Air Side

SRF - Simulated Reformer Fuel 74%. H2, 25% CO2, 1% CO

TABLE X
CELL NO LOAD POTENTIALS

350°F

Hydrogen/air

CELL NO. STACK ASS'Y 1 2 3 4 5 6 7 9 8 10 40 .84 .84 .84 .85 Dry .85 .85 .86 .86 .86 .86 41 .88 .88 .82 .89 .87 .87 .87 .87 .88 .86 Dry 42 .88 .88 .88 .88 .86 .88 .87 .88 .88 .87 Dry 43 .89 .87 .87 .87 .87 .87 .88 .88 .89 .87 Dry 44 Dry .85 .87 .83 .87 .85 .85 .84 .86 .85 .84 46 .85 .86 .87 .88 .87 .87 .87 .87 .87 .84 Wet .92 47 Wet .90 .84 .87 .86 .90 .87 .92 .90 .90 48 .86 .86 .86 .87 .87 .87 .88 .88 .88 .88 Wet 49 Wet .85 .86 .85 .86 .84 .87 .87 .86 .87 .85 50 .85 .85 .85 .85 .85 .83 .85 .86 .86 .85 Wet

TABLE XI

CELL LOAD POTENTIALS

330-350°F

40A

Hydrogen/air

CELL NO.

STACK	ASS'Y	1	2	3	4	5	<u>6</u>	7	8	9	10
40	Wet	.58	.59	.60	.60	.59	.59	.60	.59	.59	.58
41	Wet	.59	.61	.60	.62	.61	.60	.61	.60	.60	.59
42	Wet	.58	.59	.60	.59	.60	.59	.59	.59	.58	.57
43	Wet	.60	.60	.60	.60	.60	.62	.60	.61	.60	.59
44	Wet	.57	.59	.59	.60	.60	.60	.59	.60	.57	.57
46	Dry	.59	.61	.61	.62	.61	.61	.61	.61	.62	.60
47	Dry	.58	.59	.61	.60	.61	.60	.60	.61	.60	.59
48	Dry	.59	.59	.60	.60	.60	.60	.60	.60	.61	.60
49	Dry	.60	.61	.61	.61	.59	.61	.61	.61	.61	.58
50	Dry	.58	.59	.59	.60	.59	.60	.61	.59	.59	.59

FIGURE 29
STACK POLARIZATION

H₂/AIR, 350°F DRY ASSEMBLY

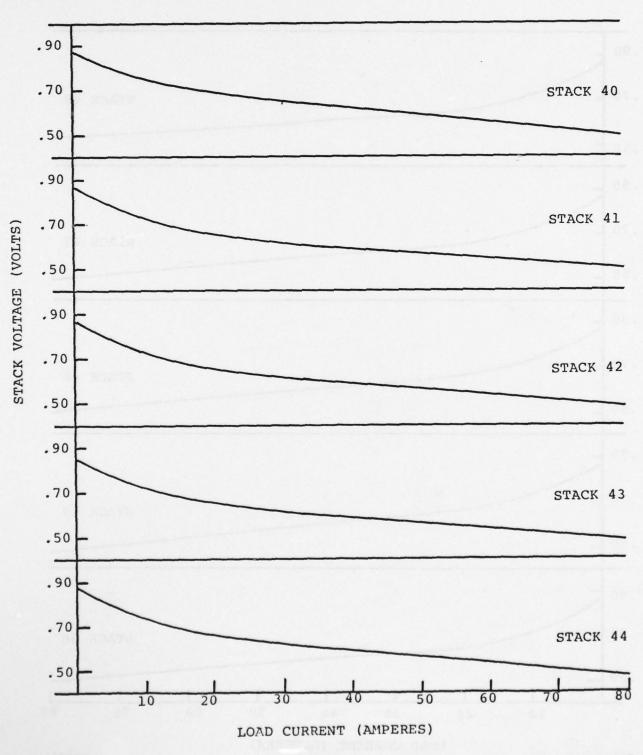


FIGURE 30

STACK POLARIZATION

H₂/AIR, 350°F WET ASSEMBLY

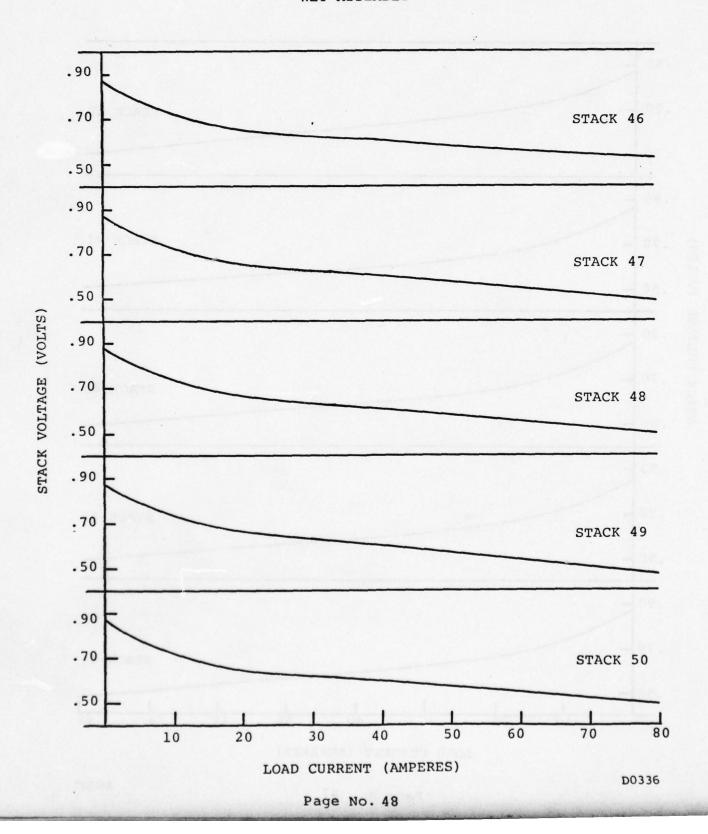


TABLE XII
STACK PERFORMANCE WITH SRF

Current: 40A (100 ASF)

Temperature: 350°F

SRF: 74% H₂, 25% CO₂, 1% CO

	Stack	42	Stac	k <u>46</u>
Cell No.	<u>H2</u>	SRF	<u>H₂</u>	SRF
1	.54	.52	.56	.54
2	.60	.58	.58	.57
3	.60	.58	.59	.58
4	.60	.58	.59	.58
5	.60	.58	.59	.58
6	.59	.57	.60	.59
7	.59	.57	.61	.59
8	.59	.57	.60	.59
9	.58	.56	.59	.58
10	.54	.52	.59	.58

FIGURE 31 STACK PERFORMANCE STABILITY (DRY ASSEMBLY)

40A 350°F

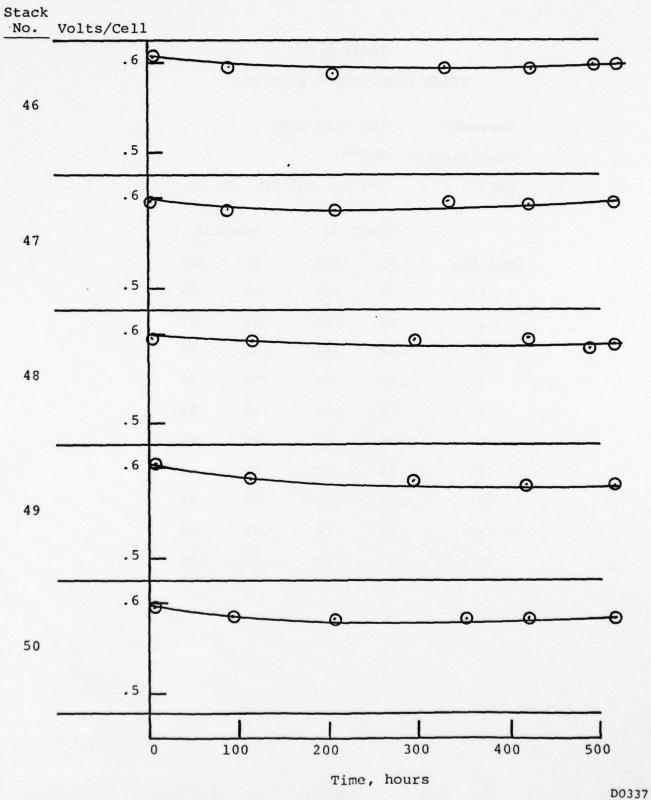
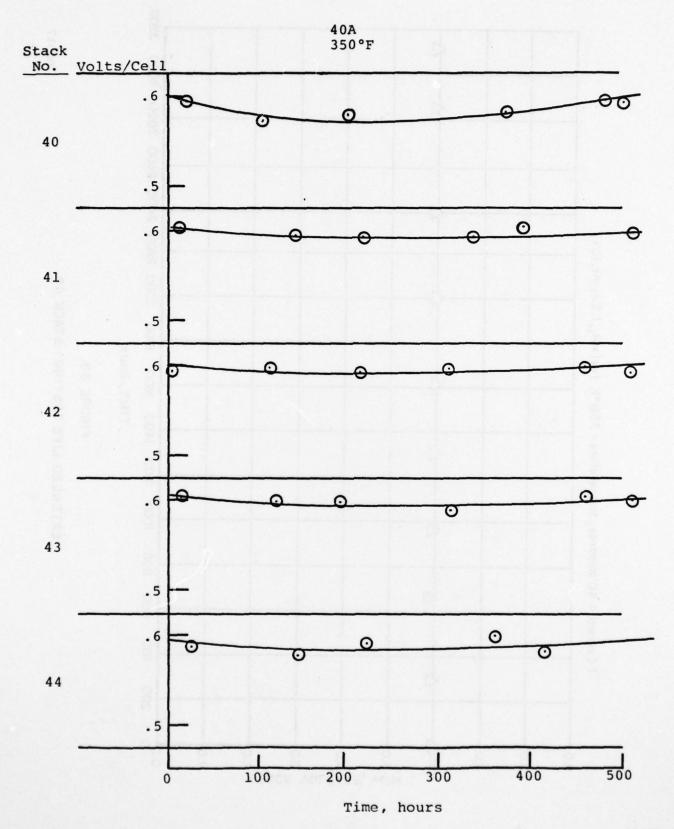
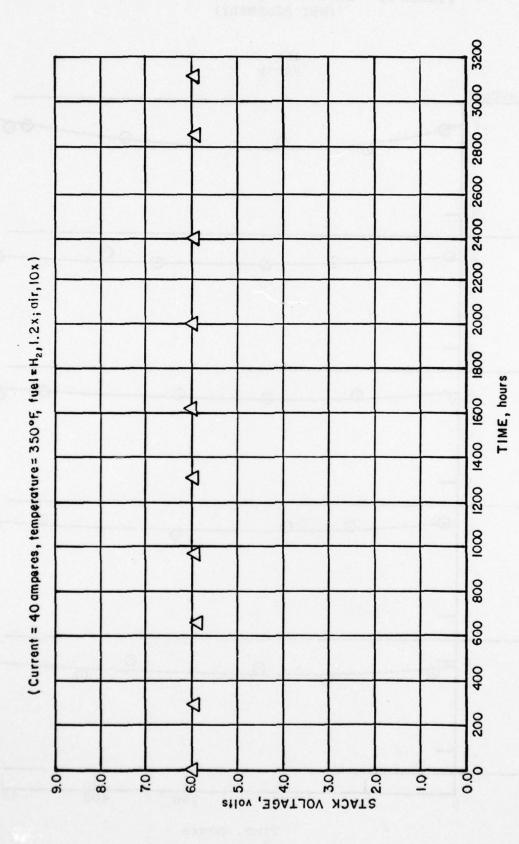


FIGURE 32 STACK PERFORMANCE STABILITY (WET ASSEMBLY)





EXTENDED LIFE TESTING - STACK 48

FIGURE 33

TABLE XIII

80 CELL STACK COMPONENTS

Cathode

Catalyst: 10% Pt on C $0.85 \pm 0.5 \text{ g/ft}^2$ Backing: Graphite paper Thickness: 0.023 \pm .002 in. Weight: 17.8 \pm 0.89

Anode

Catalyst: 10% Pt on C Catalyst Loading: 0.60 \pm .05 g/ft² Backing: Graphite paper Thickness: 0.021 \pm .02 in. Weight: 13.0 \pm 0.7g

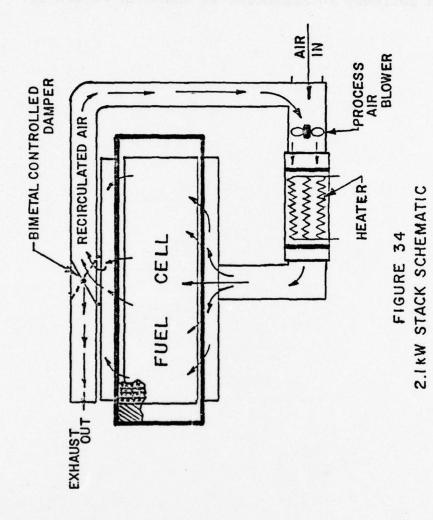
Matrix

Material: Kynol .018 \pm .011 in. Weight: 7.7 \pm 0.4g

Bipolars: "A" rib pattern fuel side
"B" rib pattern air side
33% Colloid 8440 resin, 67% graphite

TABLE XIV STACK COMPONENT WEIGHTS

	Unit Weight grams	Number	Total Weight, Kg
Bipolar Plates	192	81	15.55
Anodes	13	80	1.04
Cathodes	18	80	1.44
Matrices	8	80	.64
Ta Inserts	2	160	.32
Cement	2.5	80	.20
Electrolyte	29	80	.36
* 10000 030 81 20 1 2 91 0 7		(Subtotal	19.55)
Current Collectors	47	2	.10
Insulators	675	2	1.30
Endplates	3319	2	6.64
Air Manifolds	550	2	1.10
Fuel Manifolds	403	2	.81
Fill Ports	72	4	.29
Tie Bars	773	4	3.09
Tie Rods	252	4	1.01
		Total	33.89 Kg (74.6 lbs)



A polarization curve for the 80-cell stack obtained with fuel consisting of 80% H_2 and 20% CO_2 is shown in Figure 35. Performance of this stack was consistent with 3- and 10-cell stack performance data. The somewhat lower average cell voltages observed are due to a lower average cell temperature in the 80-cell stack. A photograph of the stack before installation of gas manifolds and with the manifolds installed (prior to delivery to MERADCOM) is shown in Figure 36.

80-CELL STACK PERFORMANCE

FIGURE 35

D0519

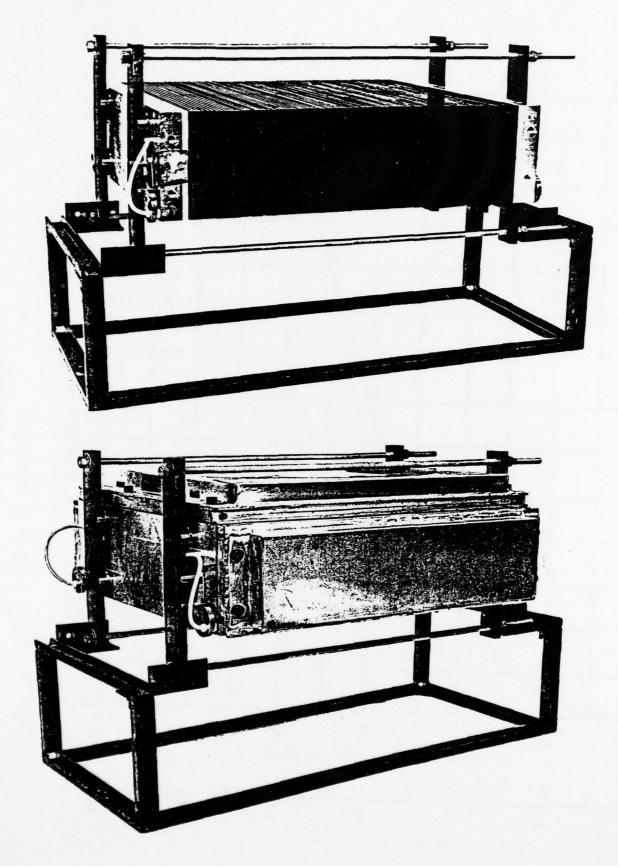


FIGURE 36

P0097

80-CELL STACK WITHOUT GAS MANIFOLDS AND WITH MANIFOLDS INSTALLED

5.0 CONCLUSIONS AND RECOMMENDATIONS

The objective of this project include improvement of cell components and stack assembly techniques for increased performance and reliability. Significant cell voltage gains have been demonstrated by the use of platinum on carbon supported catalyst while simultaneously reducing precious metal loading of the electrodes from 4-5 grams/ft² to 1-1.5 grams/ft². Tolerance to CO and stability of performance with simulated reformer product gas has been demonstrated.

Stack assembly techniques developed on this project produced reliable, gastight stacks as demonstrated by the successful consecutive assembly of ten 10-cell stacks. Endurance of the stack assembly as well as the components has been demonstrated by continuous stack operation under load. Seventeen 3-cell stacks were operated beyond 1,000 hours for a total of 49,600 hours, and eleven 10-cell stacks each logged over 1,000 hours for a total of 21,300 hours. On an individual cell basis, this represents 362,100 total cell test hours. Also, six 3-cell stacks were operated for over 4,000 hours and one for over 6,000 hours.

Future testing should include extended operation on reformed fuel as well as operation at higher temperatures and current densities. More testing of the SiC matrix is required to establish reliability of assembly and stack durability.

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